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January 28, 2000

Lara P. Autry
U. S. Environmental Protection Agency
79 T. W. Alexander Dr.
4201 Building, 4th Floor (MD-14)
Research Triangle Park, NC 27709

RE: Speciated Mercury Emissions Data

Dear Ms Autry:

Enclosed with this letter is a copy of the FINAL REPORT, MERCURY SPECIATION SAMPLING FOR THE ICR AT GREAT RIVER ENERGY, STANTON STATION UNIT 1 and the FINAL REPORT, MERCURY SPECIATION SAMPLING FOR THE ICR AT GREAT RIVER ENERGY, STANTON STATION UNIT 10. These documents serve as the final reports for the activities resulting from measuring the speciated mercury emissions at the Great River Energy's Stanton Station Units 1 and 10, as required by the EPA mercury ICR. The documents contain a plant description, sampling location information, unit operating information, descriptions of the sampling and analytical methods, quality assurance/quality control (QA/QC) activities, data from the mercury speciation sampling, and a summary and discussion of results.

Please contact me at 701 745-3387 if you have any questions or comments concerning the enclosed documents.

Sincerely,

Steve Smokey

Environmental Engineer

Enc.: Unit 1 and Unit 10 reports



FINAL REPORT MERCURY SPECIATION SAMPLING FOR THE ICR AT GREAT RIVER ENERGY STANTON STATION UNIT 1

JANUARY 2000

Prepared for:

GREAT RIVER ENERGY

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Principal Investigator – Dennis Laudal 2000-EERC-01-01

ACKNOWLEDGMENT

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1.0 INTRODUCTION

1.1 SUMMARY OF TEST PROGRAM

The U.S. Environmental Protection Agency (EPA) has implemented an Information Collection Request (ICR) aimed at characterizing mercury emissions from coal-fired power plants in the United States. As part of this ICR, the operators of selected coal-fired boilers are required to collect and analyze flue gas samples for particulate, elemental, and oxidized mercury. Flue gas samples are to be collected at the inlet of the boiler's last air pollution control device and at the unit stack using the Ontario Hydro (OH) mercury speciation method. Additionally, fuel samples are to be collected and analyzed for mercury and chlorine content.

This document serves as the final report for the activities resulting from measuring the speciated mercury emissions at Great River Energy's Stanton Station Unit 1, as required by the EPA mercury ICR. The document includes a plant description, sampling location information, unit operating information, descriptions of the sampling and analytical methods, quality assurance/quality control (QA/QC) activities, data from the mercury speciation sampling, and a summary and discussion of results.

The test program was performed to meet the requirements of the EPA mercury ICR. EPA selected the test unit based on plant configuration to provide speciated mercury emissions data, which is to be used to develop emission factors for boilers in its class. The intent of the ICR is to provide the EPA with data that can be used to develop speciated mercury emission factors for coal-fired boilers in the United States and to provide information on particulate and SO₂ control device efficiency for collecting mercury. The units to be tested have been selected on the basis of the type of coal burned, type of SO₂ control, and type of particulate control device(s).

As the owner of one of the selected units, Great River Energy, through a tailored collaboration with EPRI, the U.S. Department of Energy (DOE), and the North Dakota Industrial Commission, contracted the Energy & Environmental Research Center (EERC) to conduct the required tests at its Stanton Station.

Responsible organizations for this project are:

• Test site operator: Great River Energy (Stanton Station)

Sampling and analytical team: University of North Dakota EERC

• QA/QC oversight: RMB Consulting and Research

The test unit was Stanton Station Unit 1. This unit is operated by Great River Energy and is located near Stanton, North Dakota, in EPA Region 8. The unit was selected by EPA as part of the following category:

• Fuel type: North Dakota lignite

• SO₂ control type: none

• Particulate control type: electrostatic precipitator (ESP)

The Stanton Station consists of two pulverized coal-fired boilers. Unit 1 generates a gross output of approximately 150 MW. The Unit 1 boiler was manufactured by Foster Wheeler and is a wall-fired boiler with a gross heat input of 1800 10⁶ Btu/hr. It is equipped with an ESP to control particulate matter. The coal burned at the Stanton Station is a North Dakota lignite from the Freedom mine.

The dates of the testing were August 23–28, 1999. Measurements using the OH speciation method were completed to determine speciated mercury emissions at the inlet of the ESP (the last air pollution control device) and at the stack (outlet duct from the ESP). Fuel samples were collected at the coal feeders and analyzed for mercury and chlorine content. In addition, fly ash samples were collected from the ESP hopper and analyzed for mercury to verify the concentration of particulate-bound mercury.

The program included the following tests performed during three separate runs. Sampling was performed simultaneously at four sampling locations (inlet to the ESP, outlet from the ESP, coal feed, and ESP ash) for each run to meet the ICR requirements. Note: the ESP ash samples were collected to verify the particulate mercury concentration and were not directly required for the ICR.

In addition, mercury CEMs (continuous emission monitors) were operated at the stack location. The CEMs were operated to verify the gas-phase mercury concentration at the stack location, but were not directly required for the ICR.

Samples were taken to generate the following data:

- Particulate-bound, oxidized, and elemental mercury emissions at the ESP exhaust duct using the OH speciation method.
- Particulate-bound, oxidized, and elemental mercury concentrations at the inlet to the ESP using the OH mercury speciation method.
- Mercury and chlorine content of representative coal samples collected from the coal feeders simultaneously with the OH mercury speciation method sampling.
- Mercury content of a composite fly ash sample collected from the ESP hoppers simultaneously with the OH mercury speciation method sampling.
- Gas-phase mercury concentrations at the stack location using mercury CEMs concurrent with the OH mercury speciation method sampling.

1.2 KEY PERSONNEL

Table 1-1 lists the test program organization and key individuals with responsibilities, phone numbers, and e-mail addresses. Table 1-2 shows the responsibilities assigned to each position.

The Principal Investigator for the project is Mr. Dennis Laudal from the EERC. He reports directly to Mr. Paul Chu, Project Manager, of EPRI.

The Project QA Manager is Mr. Dave Brekke of the EERC, who is also the EERC's QA Manager. Mr. Brekke has no specific project technical assignments other than QA. As Project QA Manager, he was independent and reported only to the Office of the Director at the EERC.

Mr. Ralph Roberson of RMB Consulting and Research provided outside QA services under separate direct contract with EPRI. Activities included review of test plans and procedures as well as

TABLE 1-1
TEST PROGRAM ORGANIZATION AND RESPONSIBILITIES

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Organiza	Organization Individual	Responsibility	Reports To	Phone Number	Fax Number	E-mail Address
Plant Reg	Plant Representative				1	
GRE	Steve Smokey	Environmental Engineer NA	NA	(701) 745-3387	(701) 745-3312	ssmokey@grenergy.com
Project N	Project Management					
EPRI	Paul Chu	Project Manager		(650) 855-2812	(650) 855-2002	pchu@epri.com
DOE	Tom Brown	Project Review	NA	(412) 892-4691	(412) 892-5917	Thomas.Brown@fetc.doe.gov
EERC	Dennis Laudal	Principal Investigator	Paul Chu	(701) 777-5138	(701) 777-5181	dlaudal@eerc.und.nodak.edu
EERC Field Team	eld Team					
EERC	Richard Schulz	Field Manager	Dennis Laudal	(701) 777-5218	[(701) 777-5181	rschulz@eerc.und.nodak.edu
EERC	Rav DeWall	Project Chemist	Richard Schulz	(701) 777-5186	(701) 777-5181	rdewall@eerc.und.nodak.edu
EERC	Karen Uhrich	Assistant	Richard Schulz	(701) 777-5191	(701) 777-5181	kuhrich@eerc.und.nodak.edu
FERC	Marlvs Heidt	Sample Custodian	Richard Schulz	(701) 777-5168	(701) 777-5181	mheidt@eerc.und.nodak.edu
EERC	Jeff Thompson	Team Leader	Richard Schulz	(701) 777-5245	(701) 777-5181	jthompson@eerc.und.nodak.edu
EERC	Don Toman	Team Leader	Richard Schulz	(701) 777-5227	(701) 777-5181	dtoman@eerc.und.nodak.edu
EERC	Craig Eken	Assistant Sampler	Jeff Thompson	(701) 777-5000	(701) 777-5181	
FERC	Ray Johnson	Assistant Sampler	Don Toman	(701) 777-5000	(701) 777-5181	-
FERC	Grant Dunham	Assistant Sampler	Richard Schulz	(701) 777-5034	(701) 777-5181	gdunham@eerc.und.nodak.edu
EERC	Steve Evanson	Assistant Sampler	Richard Schulz	(701) 777-5000	(701) 777-5181	
OA/OC						
RMB	Ralph Roberson	Ralph Roberson QA/QC Manager	NA	(919) 510-0376	(919) 510-5104	roberson@rmb-consulting.com
EERC	Dave Brekke	EERC QA Manager	EERC Director	(701) 777-5154	(701) 777-5181	dbrekke@eerc.und.nodak.edu

TABLE 1-2
TEST PERSONNEL AND RESPONSIBILITIES

Staff Assignment	Responsibilities
1. Project Manager	Maintains contact with EPA as to requirements and provides updates on any new information. Helps to maintain communication between plant representative and test participants. Reviews data and input on all reports. Assists in other activities as required.
2. Principal Investigator	Coordinates all test activities. Maintains communication between all test participants. Maintains custody of data sheets and reduced data. Reviews all data. Prepares necessary reports. Assists in other activities as required.
3. Field Manager	Coordinates or performs all sample train loading and recovery activities. Maintains sample custody records. Ships samples to laboratory. Assists in other activities as required. Also coordinates or performs all sample train recovery and analytical activities. Maintains sample custody records. Transfers custody of samples to on-site laboratory. Assists in other activities as required.
4. Sample Team Leaders	Prepare and operate Ontario Hydro trains. Record and reduce data. Assist in sample recovery and other activities as required.
5. Sampling Assistants	Assist in preparation and operation of Ontario Hydro trains. Assist in sample recovery and other activities as required.
6. Project Chemist Performs all analytical activities at on-site laboratory. Ma sample custody records. Ships samples to off-site laboratory necessary.	
7. Sample Custodian	Maintains sample custody records. Transfers samples to on-site laboratory. Assists in sample recovery and other activities as required.

provision of blind spike materials for analysis. Mr. Tom Brown from DOE also provided technical review for the project. Figure 1-1 shows the organizational chart.

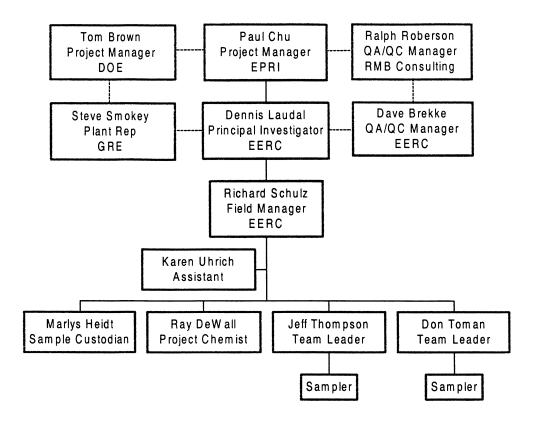


Figure 1-1. Project organizational chart.

2.0 SOURCE AND SAMPLING LOCATION DESCRIPTIONS

2.1 PROCESS DESCRIPTION

The Stanton Station consists of two pulverized coal-fired boilers. Both Units 1 and 10 burn North

Dakota lignite to generate steam for electricity production. Unit 1 generates a gross output of

approximately 150 MW. The Unit 1 boiler was manufactured by Foster Wheeler and is a wall-fired

boiler with a gross heat input of 1800 10⁶ Btu/hr. The flue gas from the Unit 1 boiler is ducted to two

ESPs (east and west in a parallel configuration) for particulate removal before being ducted to the

stack. Figure 2-1 shows a schematic of the Unit 1 boiler, ESP, and stack, including flue gas sampling

locations.

Key unit parameters include the following:

• Unit capacity: approximately 150 MW gross

Boiler type: wall fired

Fuel type: North Dakota lignite

SO₂ control: none

Particulate control: ESP

• NO_x control: low-NO_x burners

Fuel samples were collected from the three coal feeders ahead of the boiler; inlet samples were

collected at the inlet to the ESP; and stack samples were collected at the outlet from the ESP. In

addition, ash was collected from the ESP hopper, and mercury CEMs were operated at the stack

location. The stack samples were collected at the ESP exhaust duct because the stack is common to

Units 1 and 10 and there was no way to separate the flows at the stack. Unit operation during testing

was at or near nominal full load at steady-state operation. Coal type, boiler operation, and ESP

operation were all within normal operating ranges.

2-1

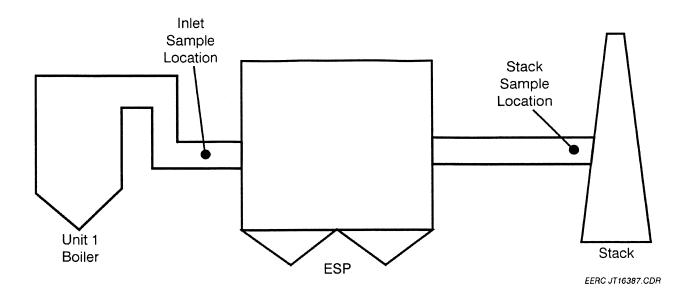


Figure 2-1. Schematic of Stanton Station Unit 1.

2.2 CONTROL EQUIPMENT DESCRIPTION

Particulate control for the Unit 1 boiler is accomplished using two parallel ESPs. Each of the ESPs has a nominal specific collection area of 470 ft² collection area/1000 ft³ gas (at 165°C [329°F]), and collection efficiency is greater than 99.9%. The split in the flue gas for the east and west ESPs is equally proportional (50:50). The east ESP was used for sampling to meet the requirements for the ICR.

2.3 FLUE GAS SAMPLING LOCATIONS

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Table 2-1 presents a summary of key inlet and stack sample location parameters. Individual discussions of the sampling locations are presented below.

TABLE 2-1
SAMPLING LOCATION DESCRIPTIONS

Description	ESP Inlet	ESP Outlet
Description		
Physical Access	Stairs/ladder	Stairs/ladder
Side or Top Access	Тор	Тор
Round or Rectangular	Rectangular	Rectangular
Inside Dimensions, ft	11.5 × 10	11.5 × 10
Equivalent Diameter, ft	10.7	10.7
Number/Type of Ports	9	9
Port Length, ft	1.17	1.17
Port Diameter, ft	0.33	0.33
Nearest Upstream Disturbance	Expansion	Bend
Distance, ft	36.7	12.2
Distance, equivalent diameters	3.4	1.1
Nearest Downstream Disturbance	Bend	Bend
Distance, ft	7.7	16.3
Distance, equivalent diameters	0.72	1.5
Typical Flue Gas Conditions		
Temperature, °F	329	332
Moisture, %	12.8	13.7
Flow rate, scfm	NA	182,000
$\mathrm{O_2},\%$ dry	6.7	6.7
CO ₂ , % dry	12.9	12.7
Particulate Concentration, gr/scf	2.0626	0.0020
SO ₂ , lb/10 ¹² Btu	NA	1.56
NO_x , lb/ 10^{12} Btu	NA	0.42

2.3.1 Inlet Location

The inlet samples were collected at existing sample ports in the duct at the inlet to the ESP. A schematic and cross section of the inlet location are shown in Figure 2-2. The sampling location for the inlet to the ESP meets EPA Method 1 criteria. It is located 3.4 equivalent diameters downstream from an expansion in the ductwork and 0.72 equivalent diameters upstream from a bend in the ductwork. The sampling ports are located in a horizontal section of steel ductwork that is 10 feet

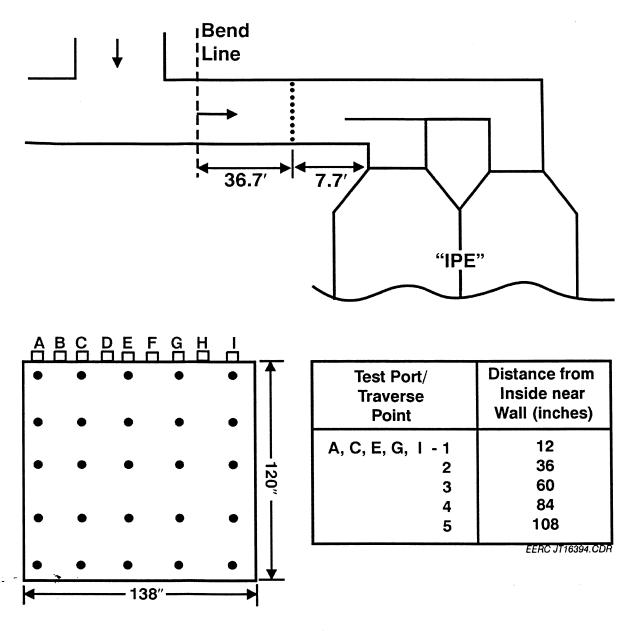


Figure 2-2. Illustration of inlet sampling location.

deep and 11.5 feet wide. Nine sample ports (A through I) are aligned on the top of the duct 36.7-feet downstream and 7.7-feet upstream of the nearest gas stream flow disturbances.

Sample traverse points for the inlet location are also shown in Figure 2-2. Five of the nine ports were used for mercury sampling. 25 traverse points with a 5×5 sample grid were used for this duct configuration. The flue gas temperature was $329^{\circ}F$, which is above the method specification of a minimum filtration temperature of $120^{\circ}C$ ($248^{\circ}F$); therefore, in-stack filtration according to Method 17 was used. This approach is considered to be consistent with the intent and data quality requirements of the ICR.

2.3.2 Stack Location

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The stack samples were collected at existing sample ports in the outlet duct of the ESP. A schematic and cross section of the stack location are shown in Figure 2-3. The sampling location for the stack does not meet EPA Method 1 criteria because the ports are located only 1.1 diameters downstream from a bend in the duct at the ESP outlet. The sampling ports are located in a horizontal section of steel ductwork that is 10 feet deep and 11.5 feet wide. Nine sample ports (A through I) are aligned on the top of the duct 12.2-feet downstream and 16.3-feet upstream of the nearest gas stream flow disturbances. Although these test ports do not meet the EPA Method 1 criteria, three-dimensional flow testing was not performed. The mercury at the stack location was (as anticipated) very near 100% in the gaseous phase and, therefore, not impacted by uncertainties in gas flow and isokinetic sampling rate.

A 5×5 traverse grid (25 traverse points) was used at the stack location. Sample traverse points for the stack location are illustrated in Figure 2-3. The flue gas at the stack was 332°F, which is above the method specification of a minimum filtration temperature of 120°C (248°F); therefore, in-stack filtration according to EPA Method 17 was used.

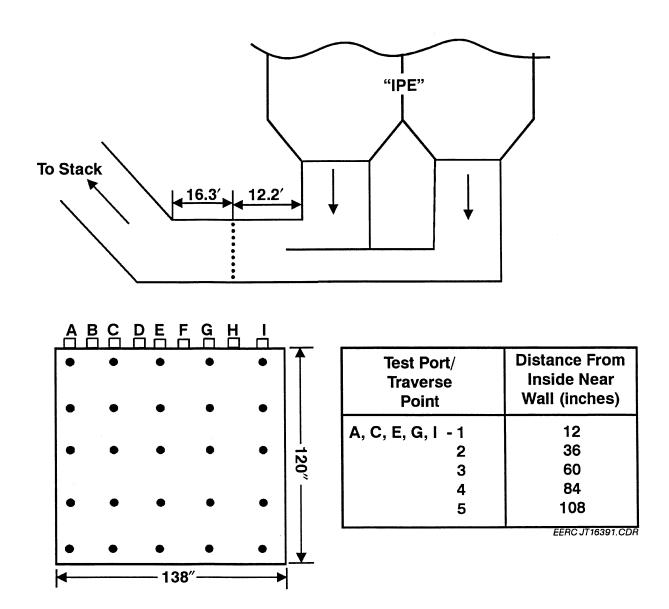


Figure 2-3. Illustration of stack sampling location.

2.4 PROCESS SAMPLING LOCATIONS

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Coal samples were collected at the coal feeders to each individual mill. These samples were collected during each of the three flue gas sampling periods. In accordance with ASTM procedures, 12 individual coal samples (four from each mill) were collected during each 2-hour sampling period. These samples were then composited prior to analysis.

Although not required by the ICR, ESP hopper ash was collected concurrently with the OH method sampling. The samples were mixed to create a composite sample for each test run.

Auxiliary gas analysis (O_2, CO_2) was done concurrently with the OH method using an additional gas sampling line in the probe.

A probe was placed in one of the ports not used for the ICR sampling, at the stack location, to sample for the mercury CEMs.

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3.0 SUMMARY AND DISCUSSION OF RESULTS

3.1 OBJECTIVES AND TEST MATRIX

The objective of the program is to collect the information and measurements required by the EPA mercury ICR. Specific objectives are to:

- Quantify speciated mercury emissions at the stack (outlet duct from the ESP).
- Quantify speciated mercury concentrations in the flue gas at the inlet to the ESP.
- Quantify fuel mercury and chlorine content during the stack and inlet tests.
- Quantify ESP hopper ash mercury content during the stack and inlet tests.
- Quantify gas-phase mercury emissions at the stack location using mercury CEMs.
- Provide the above information for use in developing boiler-, fuel-, and control device-specific mercury emission factors.

The test matrix is presented in Table 3-1. The table includes a list of test methods used. In addition to speciated mercury, the flue gas measurements included moisture, stack gas flow, and O₂/CO₂. Testing at Great River Energy's Stanton Station Unit 1 was carried out over the 2-day period of August 24–25, 1999. Table 3-2 presents the date and time information for each individual run.

3.2 FIELD TEST CHANGES AND PROBLEMS

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Field testing was carried out according to the test plan in all aspects except sampling times. Because of minor problems in the operation of the meter boxes, the sampling time was longer than anticipated at the stack during Run 1 and at the inlet during Run 3. It was decided that the additional 5 minutes beyond the 2-hour sampling time would not affect the data or the intent of the ICR; therefore, no corrective action was taken.

TABLE 3-1
TEST MATRIX FOR MERCURY ICR TESTS AT STANTON STATION UNIT 1

Sampling Location	No. of Runs	Species Measured	Sampling Method	Sample Run Time	Analytical Method
Stack	3	Speciated Hg	Ontario Hydro	~120 min	Ontario Hydro
Stack	3	Moisture	EPA 4	Concurrent	Gravimetric
Stack	3	Gas Flow	EPA 1/2	Concurrent	Pitot traverse
Stack	3	O ₂ /CO ₂	Integrated batch sample	Concurrent	Portable O ₂ /plant CEMs
Inlet	3	Speciated Hg	Ontario Hydro	~120 min	Ontario Hydro
Inlet	3	Moisture	EPA 4	Concurrent	Gravimetric
Inlet	3	Gas Flow	EPA 1/2	Concurrent	Pitot traverse
Inlet	3	O ₂ /CO ₂	Integrated batch sample	Concurrent	Portable O ₂ /plant CEMs
Coal Feeders	3	Hg, Cl in coal	Modified ASTM D2234	One grab sample per mill per run	EPA SW 846: modified 3051 (Hg) ASTM D2361, (Cl)
ESP Ash	3	Hg	Modified ASTM D2234	One composite sample per run	EPA 7473
Stack	3	Gas Phase Hg	CEMs	Concurrent	PS Analytical Sir Galahad, Semtech Hg 2000, Semtech Hg 2010

TABLE 3-2
RUN TIMES FOR MERCURY ICR TESTS AT STANTON STATION UNIT 1

Run No.	1	2	3
Date	8/24/1999	8/25/1999	8/25/1999
Starting Clock Time	1227	0935	1350
Sampling Time, min			
Inlet	125.0	125.0	130.1
Stack	130.0	125.0	125.0

3.3 PRESENTATION OF RESULTS

The results of the testing at Stanton Unit 1 are included in the following subsections:

- Process Data
- Sampling Data
- Flue Gas Mercury Data
- Mercury CEM Data
- ESP Hopper Ash Data
- Coal Analysis Data
- Mercury Mass Rates and Removal Efficiencies
- Sample Calculations

3.3.1 Process Data

The unit process data were obtained from the plant for the test periods and are summarized in Table 3-3. All data were supplied by the plant except the moisture information, which was obtained as part of the sampling activities at the stack (outlet from the ESP) location. Documentation of these data fulfills the ICR requirements and shows that the unit operation was normal during the sampling activities.

3.3.2 Sampling Data

Sampling data for each of the three runs at the inlet and stack (outlet from the ESP) are summarized in Tables 3-4 and 3-5. The tables also include the resulting calculated values according to the appropriate method (EPA Methods 2-5). The target sample volume of 1 to 2.5 m³ was obtained as well as the isokinetic sampling rate range of 90% to 100%. The calculated sample volume used for

TABLE 3-3
UNIT PROCESS DATA

Boiler			
Unit Load, MW net	140	138	133
Steam Flow, klb/hr	849	844	842
Coal Mills in Service	3	3	3
Coal Flow, lb/hr	201,000	201,000	202,000
Exit Gas Temperature, °F	298	309	320
Plant CEMs			
CO ₂ , %	11.58	11.12	11.55
SO ₂ , lb/10 ¹² Btu	1.56	1.59	1.54
NO_x , lb/ 10^{12} Btu	0.42	0.41	0.42
Opacity, %	5.50	4.90	5.10
Stack Gas Flow, scfm	187,000	172,000	188,000
ESP			
Operating Voltage (4.8.12), kV	40, 31, 27	39, 31, 28	36, 32, 29
Operating Current (4.8.12), mA	0.72, 0.47, 0.42	0.76, 0.46, 0.41	0.76, 0.43, 0.42
Gas Inlet Temperature, °F	298	309	320
Gas Outlet Temperature, °F	300	314	321
Additional Data			
O ₂ , %	2.7	2.6	2.5
H ₂ O, %	13.4	13.8	14.0

subsequent mercury calculations is shown in bold. The sampling volume was corrected to standard conditions (68°F, 29.92 in Hg, dry, and 3% O_2).

3.3.3 Flue Gas Mercury Data

The speciated mercury data for the inlet and stack locations are shown in Tables 3-6 and 3-7. The raw mercury data obtained from the analytical lab are included in the appendices. The raw data were used to calculate the absolute μg of mercury for each fraction (particulate, oxidized, and elemental)

TABLE 3-4
STANTON STATION UNIT 1 INLET SAMPLING DATA

Run No.	1	2	3
Time, min	125.0	125.0	130.1
Ts, °F	344	315	328
Vm, dcf	56.954	52.282	52.082
Tm, °F	108	99	105
Pm, in. Hg	28.36	28.19	28.13
Ps, in. Hg	27.65	27.46	27.39
An, in. ²	0.0495	0.0495	0.0495
SQRTΔP	0.502	0.480	0.469
H_2O , g	157.5	137.2	149.4
Dust, g	6.01957	6.66891	9.02006
CO ₂ , %	12.8	12.9	13.1
O ₂ , %	6.9	6.7	6.5
N ₂ + CO, %	80.3	80.4	80.4
Cm	1.00	1.00	1.00
Vmc, dcf	56.954	52.282	52.082
Vm(std), dscf	50.170	46.487	45.722
Vw(std), scf	7.426	6.470	7.045
Bws	0.1289	0.1222	0.1335
Md, lb/lb-mole	30.3	30.3	30.4
Ms, lb/lb-mole	28.7	28.8	28.7
Vs, ft/sec	36.3	34.1	33.7
I, %	102	96	95
Vm*(std), Nm ³	1.113	1.046	1.043

^{*} Corrected to 3% O₂, dry, 68°F, 29.92 in. Hg.

of the sample. The mercury speciation data along with the sample volume data were used to calculate the mercury concentration in μ g/Nm³ for each fraction. The coal feed rate and coal analysis (detailed in Section 3.3.6) were used along with the stack gas flow to calculate the emission rates for mercury in lb/10¹² Btu. Sample calculations are included in Section 3.3.8. The average and standard deviation data are included in the tables and show the consistency of the test results. The results show that, typical of the combustion of lignite coals, the mercury was primarily in elemental form.

TABLE 3-5
STANTON STATION UNIT 1 STACK SAMPLING DATA

Run No.	1	2	3
Time, min	130.0	125.0	125.0
Ts, °F	376	302	317
Vm, dcf	61.136	66.355	67.946
Tm, °F	111	102	106
Pm, in. Hg	28.43	28.24	28.19
Ps, in. Hg	27.53	27.43	27.35
An, in. ²	0.0426	0.0495	0.0495
SQRTΔP	0.665	0.605	0.630
$\mathrm{H_{2}O}$, g	175.6	199.2	206.4
Dust, g	0.01116	0.00678	0.00719
CO ₂ , %	12.8	12.5	12.7
O_2 , %	6.4	6.9	6.7
N ₂ + CO, %	80.8	80.6	80.6
Cm	1.00	1.00	1.00
Vmc, dcf	61.136	66.355	67.946
Vm(std), dscf	53.690	58.824	59.663
Vw(std), scf	8.280	9.393	9.733
Bws	0.1336	0.1377	0.1403
Md, lb/lb-mole	30.3	30.3	30.3
Ms, lb/lb-mole	28.7	28.6	28.6
Vs, ft/sec	49.2	42.8	45.1
I, %	94	97	96
Vm*(std), Nm³	1.233	1.305	1.342

^{*} Corrected to 3% O₂, dry, 68°F, 29.92 in. Hg.

3.3.4 Mercury CEM Data

Concurrently with the ICR sampling, a total of three mercury CEMs were also used to obtain gasphase mercury concentration data at the stack location. The instruments used included two Semtech

TABLE 3-6
STANTON STATION UNIT 1 INLET MERCURY DATA

Run No.	1	2	3	Average	Std. Dev.
Particulate					
μg	0.03	0.11	0.071		
μg/Nm³	0.03	0.10	0.068	0.066ª	0.038
lb/10 ¹² Btu	0.02	0.07	0.05	0.045	0.025
% Total	0.3	1.2	0.7	0.7	0.4
Oxidized					
μg	0.14	0.11	0.05		
μg/Nm³	0.12	0.10	0.04	0.09 ^a	0.04
lb/10 ¹² Btu	0.08	0.07	0.03	0.06	0.03
% Total	1.3	1.1	0.5	1.0	0.4
Elemental					
μg	10.43	8.988	9.790		
μg/Nm³	9.37	8.59	9.39	9.12	0.45
lb/10 ¹² Btu	6.54	5.71	6.81	6.35	0.57
% Total	98.4	97.7	98.8	98.3	0.6
Total					
μg/Nm³	9.52	8.80	9.50	9.27	0.41
lb/10 ¹² Btu	6.64	5.84	6.89	6.46	0.55

The variation of mercury concentration between runs was greater than 25%. Because of the low level of mercury in the particulate (less than 1% of total mercury) and the low level of oxidized mercury at the inlet (1% of total mercury), this is not considered to affect the results of the ICR speciation data.

analyzers and a PS Analytical instrument. A comparison of the gas-phase mercury concentrations measured in $\mu g/m^3$ by the mercury CEMs and gas-phase mercury concentrations determined using the OH method (not corrected to 3% O_2) are provided in Figure 3-1. The data obtained with these instruments verify the results obtained with the OH method.

TABLE 3-7
STANTON STATION UNIT 1 STACK MERCURY DATA

Run No.	1	2	3	Average	Std. Dev.
Particulate					
μg	0.0395	0.023	0.0060		
μ g/Nm ³	0.032	0.018	0.004	0.018 ^a	0.014
lb/10 ¹² Btu	0.02	0.01	0.00	0.01	0.01
% Total	0.3	0.2	0.0	0.2	0.1
Oxidized					
μg	0.42	0.45	0.48		
μ g/Nm ³	0.34	0.34	0.36	0.35	0.01
lb/10 ¹² Btu	0.24	0.22	0.25	0.24	0.02
% Total	3.6	3.6	3.6	3.6	0.0
Elemental					
μg	11.17	11.95	12.77		
μ g/Nm ³	9.05	9.15	9.51	9.24	0.24
lb/10 ¹² Btu	6.51	5.89	6.76	6.38	0.45
% Total	96.1	96.2	96.3	96.2	0.1
Total					
μg/Nm³	9.42	9.51	9.88	9.60	0.24
lb/10 ¹² Btu	6.77	6.12	7.01	6.63	0.46

The variation of mercury concentration between runs was greater than 25%. Because of the low level of mercury in the particulate (less than 1% of total mercury), this is not considered to affect the results of the ICR speciation data.

3.3.5 ESP Hopper Ash Data

ESP hopper ash was collected concurrently with the ICR runs for verification of the particulate mercury concentrations. A composite sample from each run was analyzed for mercury. The results along with the comparative OH data are shown in Table 3-8. The low level of mercury in the particulate was confirmed with these results.

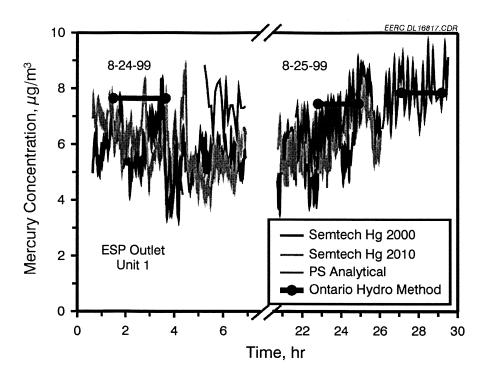


Figure 3-1. Mercury CEM data.

TABLE 3-8
ESP HOPPER ASH DATA

Run	1	2	3	Average
ESP Hopper Ash Hg, μg/g	0.013	0.0076	0.011	0.011
OH Particulate Hg, μg/g	0.005	0.016	0.0079	0.0096

3.3.6 Coal Analysis Data

The composite coal samples, one for each run, were submitted to the lab for mercury, chlorine, heating value, and proximate/ultimate analysis. The results of these analyses are summarized in Table 3-9.

TABLE 3-9
COAL ANALYSIS, 1

Mercury and Chlorine				
Mercury, ppm (dry)*	0.086	0.105	0.056	
Chlorine (Cl), ppm (dry)*	<70	<70	81	
Proximate Analysis				
Moisture, %	36.3	37.5	37.3	
Volatile Matter, %	30.89	29.94	29.71	
Fixed Carbon, %	26.58	25.71	26.70	
Ash, %	6.24	6.84	6.29	
Ultimate Analysis				
Hydrogen, %	6.55	6.73	6.71	
Carbon, %	39.37	38.71	39.04	
Nitrogen, %	0.89	0.87	0.88	
Sulfur, %	0.77	1.05	0.63	
Oxygen, %	46.18	45.79	46.45	
Heating Value				
Btu, Btu/lb	6802	6716	6716	

¹ As-received unless otherwise noted (*).

3.3.7 Mercury Mass Rates and Removal Efficiencies

Mercury flow rates were calculated for each run, in lb/hr, at each location. The results of these calculations are included in Table 3-10. For comparison, the rate at the coal feeder was divided by 2 to obtain a rate equivalent to the portion flowing to the east ESP. The results of the mass balance calculations show excellent closure for the mercury testing (average 87% from coal to stack). The removal efficiencies for each species of mercury were also calculated across the ESP. The results show that the majority of the mercury flows through the system in the gas phase unchanged. There is evidence that the ESP may be oxidizing a small portion of the mercury. Although the evidence is within the statistical limitations of the method, data from outside this project have shown that mercury can be oxidized across an ESP. The particulate-bound mercury is shown to be removed

TABLE 3-10
MERCURY MASS RATES AND REMOVAL EFFICIENCY

Run	1	2	3	Average	Std. Dev.
Mercury Balance					
Coal Feeder, lb/hr	0.0109	0.0132	0.0071	0.0104	0.0015
East Portion from Coal Feed	0.0055	0.0066	0.0035	0.0052	0.0004
Inlet, lb/hr ^a	0.0045	0.0039	0.0047	0.0044	0.0003
Stack, lb/hr ^a	0.0046	0.0041	0.0048	0.0045	
Removal Efficiency					
Particulate					
Inlet, lb/10 ¹² Btu	0.019	0.068	0.050	0.045	0.025
Stack, lb/1012 Btu	0.023	0.011	0.003	0.013	0.010
Removal, % of total	-0.1	1.0	0.7	0.5	0.5
Oxidized					
Inlet, lb/10 ¹² Btu	0.085	0.067	0.031	0.061	0.027
Stack, lb/1012 Btu	0.242	0.219	0.254	0.238	0.018
Removal, % of total	-2.4	-2.6	-3.2	-2.7	0.4
Elemental					
Inlet, lb/10 ¹² Btu	6.54	5.71	6.81	6.35	0.57
Stack, lb/1012 Btu	6.51	5.89	6.76	6.38	0.45
Removal, % of total	0.5	-3.1	0.8	-0.6	2.2
Total					
Inlet, lb/10 ¹² Btu	6.64	5.84	6.89	6.46	0.55
Stack, lb/10 ¹² Btu	6.77	6.12	7.01	6.63	0.46
Removal, %	-1.9	-4.7	-1.7	-2.8	1.7

^a Rate in one duct.

(>70%) across the ESP, but because of the small percentage of mercury in the particulate phase, the percentage of total mercury removed is minimal (0.5%).

A summary of the results from this testing with regard to mercury partitioning and emissions are as follows:

- Mercury is primarily in the elemental form (>95%).
- Particulate-bound mercury was removed across the ESP, but the percentage of total mercury removed was small (<1% of total).
- A small amount of mercury appears to be converted to the oxidized form across the ESP, but because of the small amount of oxidized mercury (<0.5% of total), results are inconclusive (within experimental error).
- Mass balance results show excellent closure for the unit.
- Mercury is not significantly removed across the ESP.

3.3.8 Sample Calculations

Sample calculations are included for each of the calculated parameters. Data from the inlet location during Run 1 were used with data from the stack location from Run 1 where necessary.

3.3.8.1 Volume of Gas Sample

Vm(std) = Volume of gas sample measured by the dry gas meter, corrected to standard

conditions, dscf

 $Vm(std) (dscf) = K_1 * Vmc * Pm / (Tm + 460)$

Vm(std) = 17.64 * 56.954 * 1 * 28.36 / (107.92 + 460) = 50.170 dscf

Where:

 K_1 = 17.64 °R/in. Hg

Vmc = Vm * Cm = Volume of gas sample as measured by dry gas meter corrected

for meter calibration (Cm = meter calibration coefficient) (dcf)

Pm = Meter pressure (in. Hg)

Tm = Meter temperature ($^{\circ}$ F)

3.3.8.2 Volume of Water Vapor

Vw(std) = Volume of water vapor in the gas sample, corrected to standard conditions,

scf

 $Vw(std) (scf) = K_2 * H_2O(g)$

Vw(std) = 0.04715 * 157.5 = 7.426 scf

Where:

 $K_2 = 0.04715 \text{ ft}^3/\text{g}$

 $H_2O(g)$ = Mass of liquid collected in impingers and silica gel (g)

3.3.8.3 Water Vapor in the Gas Stream

Bws = Water vapor in the gas stream, proportion by volume

Bws = Vw(std) / (Vm(std) + Vw(std))

Bws = 7.426 / (50.170 + 7.426) = 0.1289

3.3.8.4 Dry Molecular Weight

Md = Dry molecular weight of stack gas, lb/lb-mole

Md (lb/lb-mole) = $0.440 * (\%CO_2) + 0.320 * (\%O_2) + 0.280 * (\%N_2 + \%CO)$

Md = 0.440 * 12.8 + 0.320 * 6.9 + 0.280 * 80.3 = 30.3 lb/lb-mole

Where:

 $\%(CO_2, O_2, N_2, CO)$ = Percent (CO_2, O_2, N_2, CO) by volume, dry basis

3.3.8.5 Molecular Weight

Ms = Molecular weight of stack gas, wet basis, lb/lb-mole

Ms (lb/lb-mole) = Md * (1 - Bws) + 18.0 * Bws

Ms =
$$30.3 * (1 - 0.1289) + 18.0 * 0.1289 = 28.7$$
 lb/lb-mole

3.3.8.6 Average Stack Gas Velocity

Vs = Average stack gas velocity, ft/sec

Vs (ft/sec) = $K_3 * Cp * (\Delta p)^{1/2} avg * [(Ts + 460) / (Ps*Ms)]^{1/2}$

Vs = $85.49 * 0.84 * 0.502 * [(344 + 460) / (27.65 * 28.7)]^{1/2} = 36.3 \text{ ft/sec}$

Where:

 $K_3 = 85.49 \text{ ft/sec}[(lb/lb-mole)(in. Hg)/((°R)(in. H₂O))]^{1/2}$

Cp = Pitot tube coefficient, dimensionless

 Δp = Velocity head of stack gas (in. Hg)

Ts = Stack gas temperature (°F)

Ps = Stack pressure (in. Hg)

3.3.8.7 Isokinetic Sampling Rate

I = Percent of isokinetic sampling, %

I(%) = K_4 * (Ts + 460) * Vm(std) / (Ps * Vs * An / 144 * θ * (1 - Bws))

I = 0.09450 * (344 + 460) * 50.170 /(27.65 * 36.3 * 0.0495 /144 * 125 *

(1 - 0.1289)) = 101.5%

Where:

Ť

 $K_4 = 0.09450\% \text{ (in. Hg)(min)/((°R)(sec))}$

An = Cross-sectional area of nozzle $(in.^2)$

 θ = Total sampling time (min)

3.3.8.8 Volume of Gas Sample Corrected to $3\% O_2$

Vm*(std) = Volume of gas sample measured by the dry gas meter (Vm(std)), * corrected

to 3% oxygen, Nm³

Vm*(std) = Vm(std) * (21 - %O₂) / 18 * K₅

Vm*(std) = 50.170 * (21 - 6.9) / 18 * 0.02832 = 1.113 Nm^3

Where:

 $K_5 = 0.02832 \text{ m}^3/\text{ft}^3$

3.3.8.9 *Mercury*

Hg (μ g) = μ g/g * g or μ g/L * mL / 1000

Hg = $0.27 * 500 / 1000 = 0.135 \mu g$ (using the KCl/oxidized mercury data from

Run 1)

Hg (μ g/Nm³) = μ g / Vm*(std)

Hg = $0.135 / 1.113 = 0.121 \mu g/Nm^3$

Particulate Hg = Sum of mercury from filter and nozzle rinse (note: all nozzle rinse values

were nondetects)

Oxidized Hg = Sum of mercury from KCl impingers

Elemental Hg = Sum of mercury from H_2O_2 and $KMnO_4$ impingers

3.3.8.10 Mass Rates

Hg (lb/hr) from coal = Cf * (1 - Bc) * Hg(ppm) / 10^6

Hg (lb/hr) from coal = $201,000 * (1 - 0.3630) * 0.0855 / 10^6 = 0.109$ lb/hr

Where:

Cf = Coal feed rate (lb/hr)

Hg(ppm) = Mercury concentration in coal (ppm, dry)

Bc = Coal moisture (fraction)

Hg (lb/hr) in flue gas = Hg(μ g/Nm³) * Sf * (21 - %O₂) /18 * (1 - Bws) * K₆

Hg (lb/hr) in flue gas = 9.52 * 187,000 * (21 - 6.9) / 18 * (1 - 0.1289) * 3.745 × 10⁻⁹ = 0.0045 lb/hr

Where:

 $K_6 = 3.745 \times 10^{-9} (lb/\mu g)(m/hr)(m^3/ft^3)$

Sf = Stack gas flow (scfm)

3.3.8.11 Emission Rate

Hg (lb/10¹² Btu) = Hg (μ g/Nm³) * Sf * (1 - Bws) * (21 - %O₂) /18 /Cf /Hv * K₇

Hg (lb/ 10^{12} Btu) = 9.52 * 187,000 * 2 * (1 - 0.1289) * (21 - 6.9) /18 /201,000 /6802 * 3745

 $= 6.65 \text{ lb/}10^{12} \text{Btu}$

Where:

 $K_7 = 3745 \text{ (m}^3/\text{ft}^3)(\text{m/hr})(\text{lb/}\mu\text{g})(\text{Btu/}10^{12} \text{ Btu})$

Hv = Heating value (Btu/lb)

2 = Factor to account for both east and west ESP ducts

4.0 SAMPLING AND ANALYTICAL PROCEDURES

4.1 TEST METHODS

4.1.1 Flue Gas Mercury Speciation

This section contains a summary of the sampling and analytical procedures used to conduct the mercury speciation method required in EPA's ICR entitled Standard Test Method for Elemental, Oxidized, Particle-Bound and Total Mercury in Flue Gas Generated from Coal-Fired Stationary Sources (Ontario Hydro Method) dated May 1999. For this method, a sample is withdrawn from the flue gas stream isokinetically through the filtration system, which is followed by a series of impingers in an ice bath. Particulate-bound mercury is collected on the front half and filter; oxidized mercury is collected in impingers containing 1 N potassium chloride solution; and elemental mercury is collected in one impinger containing a 5% nitric acid and 10% peroxide solution and in three impingers containing a solution of 10% sulfuric acid and 4% potassium permanganate. An impinger containing silica gel collects any remaining moisture. Quartz fiber filters were used as the filter media for the testing, and the filter holder was glass. A heated Teflon line was used between the probe and impinger train. A target sampling time of 2 hours was used, with a target sample volume of 1 to 2.5 standard cubic meters. A schematic of the sample train is shown in Figure 4-1. Table 4-1 presents a list of sample train components for the EPA Method 17 configuration. Because the flue gas temperature was greater than 120°C (248°F) at both the inlet and stack sampling locations, instack filtration (EPA Method 17 configuration) was used.

Figure 4-2 is a schematic of the sample recovery procedure for the impinger train. The samples were recovered into precleaned glass bottles with vented Teflon-lined lids for submission to the laboratory. The following sample fractions were recovered (specific rinse solutions are contained in the method):

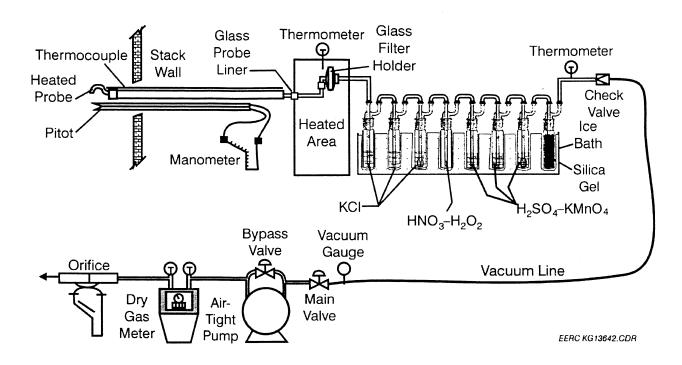


Figure 4-1. Schematic of mercury speciation sample train (Method 5 option is shown; Method 17 in-stack is also allowed).

TABLE 4-1
SAMPLE TRAIN COMPONENTS – EPA METHOD 17 CONFIGURATION

Component	Details
Nozzle	Quartz
Filter	Quartz, in glass
Probe	Quartz heated to a minimum temperature of 120°C
Connector Line	Teflon line used to connect from probe to impingers. Heated to a minimum of 120°C
Impingers 1 and 2	1 N KCl solution; modified Smith Greenburg (SG) impinger
Impinger 3	1 N KCl solution; standard SG impinger
Impinger 4	5% nitric acid-10% hydrogen peroxide; modified SG impinger
Impingers 5 and 6	10% sulfuric acid-4% potassium permanganate; modified SG impinger
Impinger 7	10% sulfuric acid-4% potassium permanganate; standard SG impinger
Impinger 8	Silica gel; modified SG impinger

- 1. Rinse filter holder and connector with 0.1N HNO₃.
- 2. Add 5% W/v KMnO4 to each impinger bottle until purple color remains.
- 3. Rinse with 10% $^{4}/_{v}$ HNO₃.
- 4. Rinse with a very small amount of 10% w/, NH₂OH·H₂SO₄ if brown residue remains.

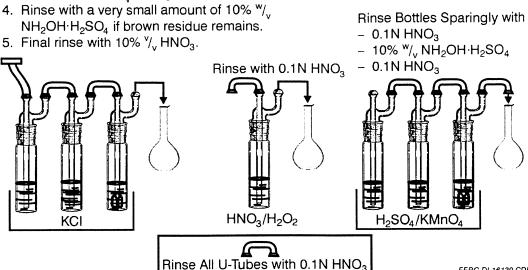


Figure 4-2. Sample recovery scheme for the mercury speciation sampling train.

EERC DI 16139 CDR

- Container 1 the sample filter
- Container 2 the front half rinse (includes all surfaces upstream of the filter)
- Container 3 Impingers 1 through 3 (KCl impingers) and rinses
- Container 4 Impinger 4 (HNO₃– H_2O_3 impinger) and rinses
- Container 5 Impingers 5 through 7 (H₂SO₄–KMnO₄ impingers) and rinse
- Silica Gel Impinger 8 (silica gel impinger). Note that this sample is weighed for moisture determination and is not included in the mercury analysis.

The sample fractions were prepared and analyzed as specified in the method and summarized below:

• Ash Sample (Containers 1 and 2) – The particulate catch was digested and analyzed using EPA Method 3051 with subsequent analysis using EPA Method 7471A. When the particulate catch was greater than 1 g (as was the case at the ESP inlet location), an aliquot of the particulate collected on the filter was analyzed. When the particulate catch was less than 1 g and an aliquot could not be taken for analysis, the entire filter and particulate catch was digested and analyzed.

- KCl Impingers (Container 3) The impingers were prepared using H₂SO₄, HNO₃, and KMnO₄ solutions as specified in the method.
- HNO_3 – H_2O_2 (Container 4) The impinger solution was prepared using HCl and KMnO₄ solutions as specified in the method.
- H_2SO_4 $KMnO_4$ Impingers (Container 5) The impinger solution was prepared using hydroxylamine sulfate as specified in the method.

Each prepared fraction was analyzed for total mercury by cold-vapor atomic absorption (CVAAS). CVAAS is a method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Mercury concentration is proportional to the indicated absorbance. A soda-lime trap and a magnesium perchlorate trap were used to precondition the gas before it entered the absorption cell.

4.1.2 Fuel Mercury and Chlorine

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Mercury in coal was determined by digesting the coal with nitric and hydrochloric acid in sealed high-pressure Teflon digestion vessels similar to EPA Method SW846 3051, Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils. The modifications to the method include 1) the use of larger, high-pressure Teflon vessels, designed specifically for coal; 2) the use of nitric and hydrochloric acid for digestion; and 3) the use of multiple cooldown and venting steps to completely digest the coal. The digested samples were analyzed by CVAAS according to EPA Method 7471A. All values were reported as $\mu g/g$ Hg on a dry coal basis.

Chlorine was determined by igniting a weighed coal sample in a combustion bomb containing oxygen under pressure in the presence of an alkaline solution, according to ASTM Method D2361, Standard Test Method for Chlorine in Coal. The solution was analyzed by ion chromatography for chloride and was reported as $\mu g/g$ Cl on a dry coal basis.

4.1.3 Auxiliary Flue Gas Measurements

EPA methods for auxiliary flue gas measurements included flue gas flow rate using EPA Methods 1 and 2 (pitot traverse), O₂ and CO₂ by EPA Method 3A (portable O₂ analyzer), and moisture by EPA Method 4 (condensation/gravimetric analysis). These measurements were collected as integral parts of all mercury speciation test runs at both the inlet and stack locations. EPA Reference Method 5/17 requirements were followed for isokinetic sampling.

4.1.4 Mercury CEM Measurements

4.1.4.1 PS Analytical Sir Galahad

The Sir Galahad analyzer was initially used to monitor total mercury continuously in the urban environment and in natural gas, but it can also be used in a variety of gaseous media including combustion flue gas. The analyzer is based on the principle of atomic fluorescence which provides an inherently more sensitive signal than atomic absorption. The system uses a gold-impregnated silica support for preconcentrating the mercury and separating it from potential interferences that degrade sensitivity.

The Sir Galahad requires a four-step process to obtain a flue gas mercury measurement. In the first step, 2 L of flue gas are pumped through a gold trap which is maintained at a constant temperature. Before the mercury is desorbed from the gold trap, a flushing step is initiated to remove any flue gas that may be present because it has a damping effect on the mercury fluorescence. When this is completed, the analysis step begins. The heating coil is activated, and the gold trap is heated to approximately 500°C. This desorbs the mercury from the trap, and the mercury is carried into the fluorescence detector. The gold trap is rapidly cooled by pumping argon over it, in preparation for the next sample. The total time for the entire process is about 5 minutes.

The system was calibrated using Hg⁰ as the primary standard. The Hg⁰ is contained in a closed vial which is held in a thermostatic bath. The temperature of the mercury is monitored, and the amount

of mercury is calculated using vapor pressure calculations. Typically, the calibration of the unit has proven stable over a 24-hr period.

4.1.4.2 Semtech Hg 2000 and Hg 2010

The commercial Semtech Hg 2000 and Hg 2010 mercury analyzers (Semtech Metallurgy AB, Lund, Sweden) are essentially portable Zeeman-modulated CVAAS instruments that can monitor Hg⁰ continuously. The flue gas was converted and conditioned with a separate unit, and the conditioned dry gas was then analyzed using the Semtech Hg 2000 analyzer. The analyzer uses Zeeman effect background correction, by applying a modulated magnetic field to a mercury lamp, to minimize interferences from the presence of SO_2 , hydrocarbons, and fine particulate in the flue gas sample. The operating range of the analyzer is $0.3 \ \mu g/Nm^3$ to $20 \ mg/Nm^3$ Hg⁰, as specified by Semtech Metallurgy AB. The Semtech Hg 2000 has also been certified by TUEV Rheinland for determining compliance with the German legal limit of $50 \ \mu g/Nm^3$ for total mercury from waste incinerators.

4.2 PROCESS TEST METHODS

The operational data collected are listed in Table 4-2. To the fullest extent possible, the data were collected using existing plant instrumentation and computerized log printouts. The objective of the process data collection was to ensure and document normal boiler and air pollution control device operation. Prior to and during each test, unit operation was assessed by the sampling team process monitor in conjunction with station personnel to ensure that operating conditions were within project target ranges.

TABLE 4-2 PROCESS DATA COLLECTED

Boiler Data

Unit Load, MW net Steam Flow, klb/hr Number of Coal Mills in Service Coal Flow, tons/hr Exit Gas Temperature, °F

CEM Data

CO₂, % wet or dry SO₂, lb/10¹² Btu NO_x, lb/10¹² Btu (record NO₂ if available) Opacity, % Stack Gas Flow, klb/hr

Electrostatic Precipitator

Operating Voltage, kV Operating Current, mA Gas Inlet Temperature, °F Gas Outlet Temperature, °F

5.0 QA/QC ACTIVITIES

Table 5-1 summarizes the Data Quality Objectives and Results for accuracy, precision, and completeness for flue gas mercury analyses (OH samples). Table 5-2 presents the evaluation and verification checklist.

TABLE 5-1

DATA QUALITY OBJECTIVES AND RESULTS FOR FLUE GAS

MERCURY ANALYSES

Measure	Activity	Objective	Result
Accuracy	Reagent blanks – one blank per batch of each reagent	<10% of sample value or <10× instrument detection limit	All reagent blanks were less than the detection limit.
Accuracy	Field blanks – one set per location (inlet, stack) per day	<30% of sample value	All field blanks were less than the detection limit.
Accuracy	Blind reagent spikes and certified reference ash sample	±10% of value	All results from spikes (internal and external) and certified reference materials meet the ± 10% criteria.
Precision	Triplicate analyses	±10% of mean	All triplicates were within 10% of means.
Completeness	Any failed or incomplete test will be reviewed and, if necessary, repeated.	100% complete	All tests were completed successfully.

TABLE 5-2
RESULTS EVALUATION AND VERIFICATION CHECKLIST

	Result	
No unusual conditions	No unusual conditions	
No unusual conditions	No unusual conditions	
<0.02 cfm	<0.02 cfm	
Zero leakage	Zero leakage	
Minimum 120°C	NA Method 17 used	
90%–110%	90%-110%	
1 to 2.5 standard cubic meters	1 to 2.5 standard cubic meters	
Purple	Purple	
All runs w/in 10% of mean (adjusted for load if necessary)	All outlet runs w/in 3% of mean. The inlet runs were more variable, w/in 5% of mean. This is not considered to affect the mercury speciation data, nor the intent of the ICR.	
All runs w/in 3% of mean	All runs w/in 3% of mean	
All runs w/in 5% of mean	All runs w/in 5% of mean	
All runs w/in 35% of mear	All runs w/in 35% of mean	
All runs w/in 25% of mear	The low level of mercury in the particulate (less than 1% of total mercury) resulted in greater variation than 25% of mean at both the inlet and the stack location. The particulate data was flagged.	
	The low level of oxidized mercury at the inlet (1% of total mercury) resulted in greater variation than 25% of mean at the inlet location. The data were flagged. The oxidized mercury at the stack location was w/in 25% of mean.	
	No unusual conditions <0.02 cfm Zero leakage Minimum 120°C 90%—110% 1 to 2.5 standard cubic meters Purple All runs w/in 10% of mean (adjusted for load if necessary) All runs w/in 3% of mean All runs w/in 5% of mean All runs w/in 35% of mear All runs w/in 25% of mear All runs w/in 25% of mear	

5.1 ACCURACY

Three indicators were used for accuracy. A reagent blank was taken from each batch of reagents prepared. The objective was <10% of the sample values or <10 times the instrument detection limit. One field blank was collected at each sample location. The field blank consisted of a sample train that was assembled, taken to the same location as a test sample, and recovered. The quality objective for a field blank was less than 30% of the typical sample values. Two blind reagent spikes and a certified reference ash sample were analyzed as part of the analytical procedure. The blind spikes were provided by RMB Consulting and Research. The objective was ±10% of the true or certified value. Results for the blind spikes were sent to RMB Consulting and were reported in an audit report. A copy of the report can be obtained from RMB Consulting. Certified reference materials are routinely analyzed by the EERC lab, and results can be obtained upon request. All accuracy criteria were met for this test.

Tables 5-3 and 5-4 show the results for the analysis of reagent blanks and field blanks.

In addition, analytical spikes were completed as an internal check for accuracy. The results of the spikes are shown in Table 5-5. All recoveries were within $\pm 10\%$.

TABLE 5-3
REAGENT BLANK ANALYSIS RESULTS

Reagent	Hg, μg/L
KCl Reagent Blank	< 0.03
H ₂ O ₂ Reagent Blank	<0.03
KMnO₄ Reagent Blank	< 0.03
5% KMnO₄ Blank	<0.03
10% HNO ₃ Blank	< 0.03
0.1 N HNO3 Blank	< 0.03

TABLE 5-4
FIELD BLANK ANALYSIS RESULTS

Sample ID	Sample Type	Volume, mL	Hg, μg/L
GRE-U1-D1-FB-IN	KCl	500	<0.03
GRE-U1-D1-FB-IN	H_2O_2	250	<0.03
GRE-U1-D1-FB-IN	KMnO₄	500	<0.03
GRE-U1-D1-FB-OUT	KCl	500	<0.03
GRE-U1-D1-FB-OUT	$\mathrm{H_2O_2}$	250	<0.03
GRE-U1-D1-FB-OUT	KMnO₄	500	<0.03
GRE-U1-D2-FB-IN	KCl	500	<0.03
GRE-U1-D2-FB-IN	H_2O_2	250	<0.03
GRE-U1-D2-FB-IN	$KMnO_4$	500	<0.03
GRE-U1-D2-FB-OUT	KCl	500	<0.03
GRE-U1-D2-FB-OUT	H_2O_2	250	<0.03
GRE-U1-D2-FB-OUT	KMnO₄	500	<0.03

5.2 PRECISION

The precision target for the program was ±10%. Every tenth sample was analyzed in triplicate. The site-specific test plan called for duplicate analyses of the liquid samples from the Ontario Hydro method for mercury. Because of time limitations in the field and the desire to analyze all the samples at the site, all of the samples were not analyzed in duplicate in the field. A request for a deviation from the test plan was accepted to eliminate the need for duplicate analyses of all of the samples. The precision of mercury analysis using CVAA has been demonstrated and documented using duplicate sample analyses for over 2 years at the EERC. Since the precision of the method has been demonstrated, the samples are no longer routinely analyzed in duplicate. The triplicate analysis of every tenth sample continues to be done to ensure instrument precision. The results of the triplicate analyses for the Stanton Station Unit 1 testing are shown in Table 5-6. In all cases, the data fall within ±10% of the mean.

TABLE 5-5
ANALYTICAL SPIKES

Sample ID	Туре	Spike Amount, µg/L	Sample Value, µg/L	Spike Reading, µg/L	Recovery,
GRE-U1-D1-IN-OH-1	KCl	5	0.27	4.93	93.20
GRE-U1-D1-IN-OH-1	KCl	10	0.27	9.7	94.32
GRE-U1-D1-FB-SPIKE	KCl	10	0	10.36	103.60
GRE-U1-D1-FB-SPIKE	KCl	5	10.36	15.68	106.40
GRE-U1-D1-FB-SPIKE	KCl	10	10.36	20.44	100.80
GRE-U1-D1-IN-OH-1	H_2O_2	5	1.29	6.39	102.00
GRE-U1-D1-IN-OH-1	H ₂ O ₂	10	1.29	11.12	98.30
GRE-U1-D1-FB-SPIKE	H_2O_2	2	0	1.92	96.00
GRE-U1-D1-FB-SPIKE	H ₂ O ₂	5	1.92	6.74	96.42
GRE-U1-D1-FB-SPIKE	H ₂ O ₂	10	1.92	11.4	94.83
GRE-U1-D1-FB-SPIKE	KMnO ₄	10	0	9.68	96.80
GRE-U1-D1-FB-SPIKE	KMnO ₄	5	9.68	14.44	95.20
GRE-U1-D1-IN-OH-1	KMnO ₄	5	19.97	24.48	90.20

5.3 COMPLETENESS

All samples were completed and verified by the sampling manager for this test.

TABLE 5-6
RESULTS FROM TRIPLICATE ANALYSES

Sample ID	Sample Type	Hg Concentration, μg/L		
GRE-U1-D1-IN-OH-1	KCl Nos. 1 and 2	0.28	0.26	0.26
GRE-U1-D1-IN-OH-1	$\mathrm{H_2O_2}$	1.35	1.23	1.3
GRE-U1-D1-IN-OH-1	KMnO_4	19.76	19.96	20.2
GRE-U1-D1-FB-SPIKE	KMnO₄	9.67	9.74	9.63
GRE-U1-D2-IN-OH-1	KMnO₄	16.6	16.75	16.83
GRE-U1-D2-STACK-OH-1	KCl Nos. 1 and 2	0.9	0.89	0.9
GRE-U1-D2-STACK-OH-1	H_2O_2	0.57	0.55	0.62